

(12) UK Patent Application (19) GB (11) 2 335 201 (13) A

(43) Date of A Publication 15.09.1999

(21) Application No 9804907.5

(22) Date of Filing 10.03.1998

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(51) INT CL⁶
C03C 17/34 , C23C 14/08 16/40 , E06B 3/66

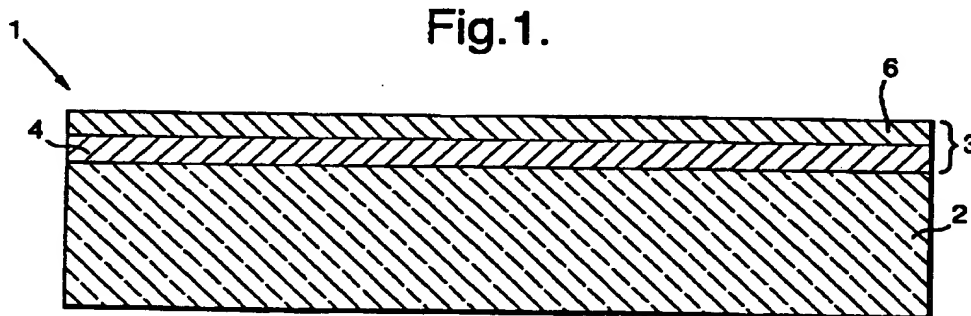
(52) UK CL (Edition Q)
C7F FBAL FHB FHE FPD L FQ909 FQ912 FQ914 FR842
FR912 FR914 F103 F109 F515 F519 F782
E1R RPP
U1S S1714 S3072 S3077

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EP 0811583 A1 WO 98/11031 A1 US 5776603 A
US 5756192 A US 5348805 A US 5342676 A
US 5244692 A US 4507547 A US 4419386 A
US 4206252 A

(58) Field of Search
UK CL (Edition Q) C7F FBAL FBAX FBBL FBBX FBXL
FBXX FHB FHD FHE FHX FPCL FPCX FPD L FPD X
INT CL⁶ C03C 17/34 , C23C 14/08 16/40
Online: WPI, EPODOC, JAPIO

(54) Abstract Title
Glass coated with two doped oxide layers

(57) A low emissivity coated glass comprises a glass substrate with a coating comprising a first layer of a doped metal oxide in combination with a second layer of a doped metal oxide; 4, 6; characterised in that the host metal oxides in each of the said layers are different. Preferably the first layer of a doped metal oxide comprises a layer of doped tin oxide, and the second layer comprises indium tin oxide or doped zinc oxide. A coated glass according to this invention will usually have a normal emissivity of 0.15 or lower. The layers may be deposited by sputtering or by chemical vapour deposition.



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At least one drawing originally filed was informal and the print reproduced here is taken from a later filed formal copy.

Fig.1.

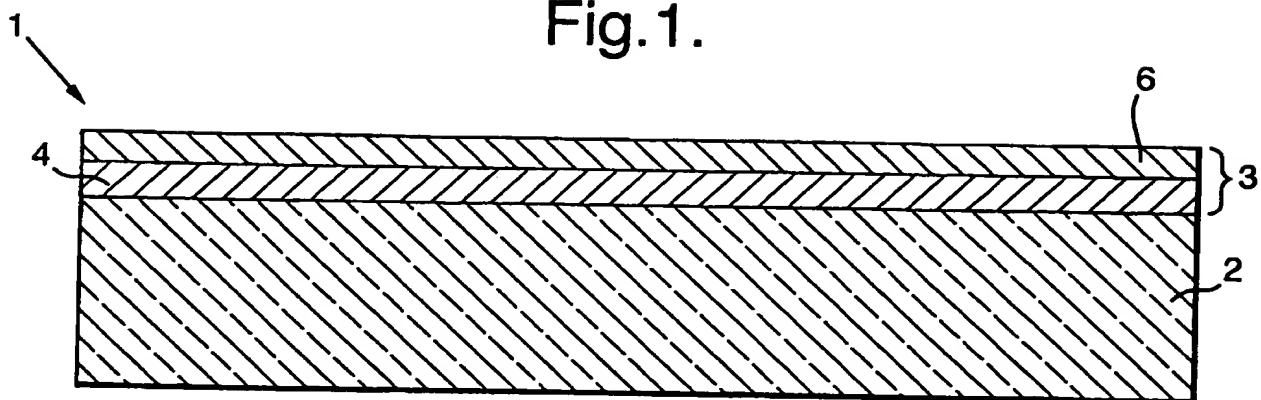


Fig.2.

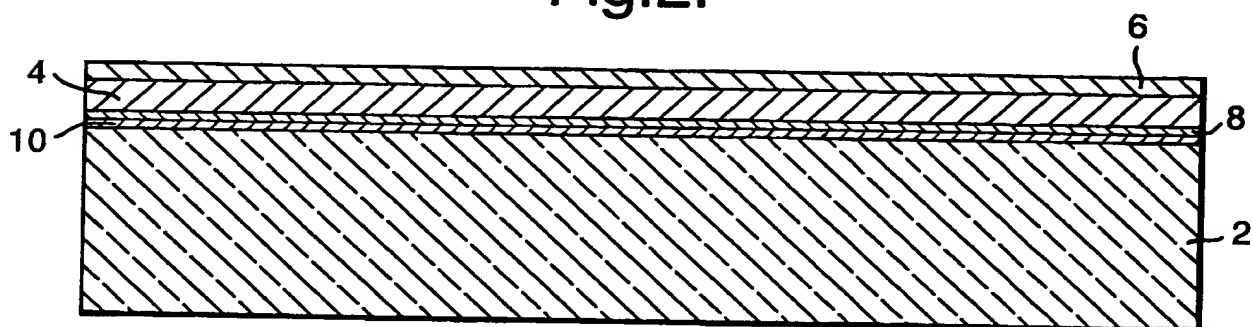
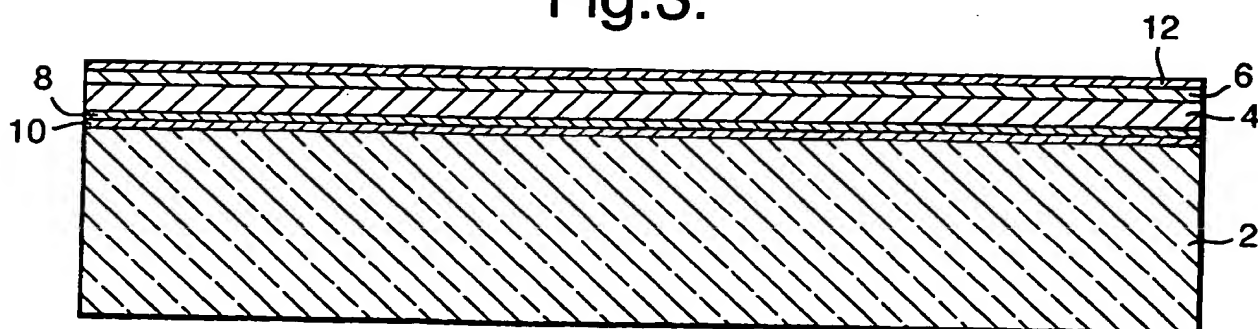


Fig.3.



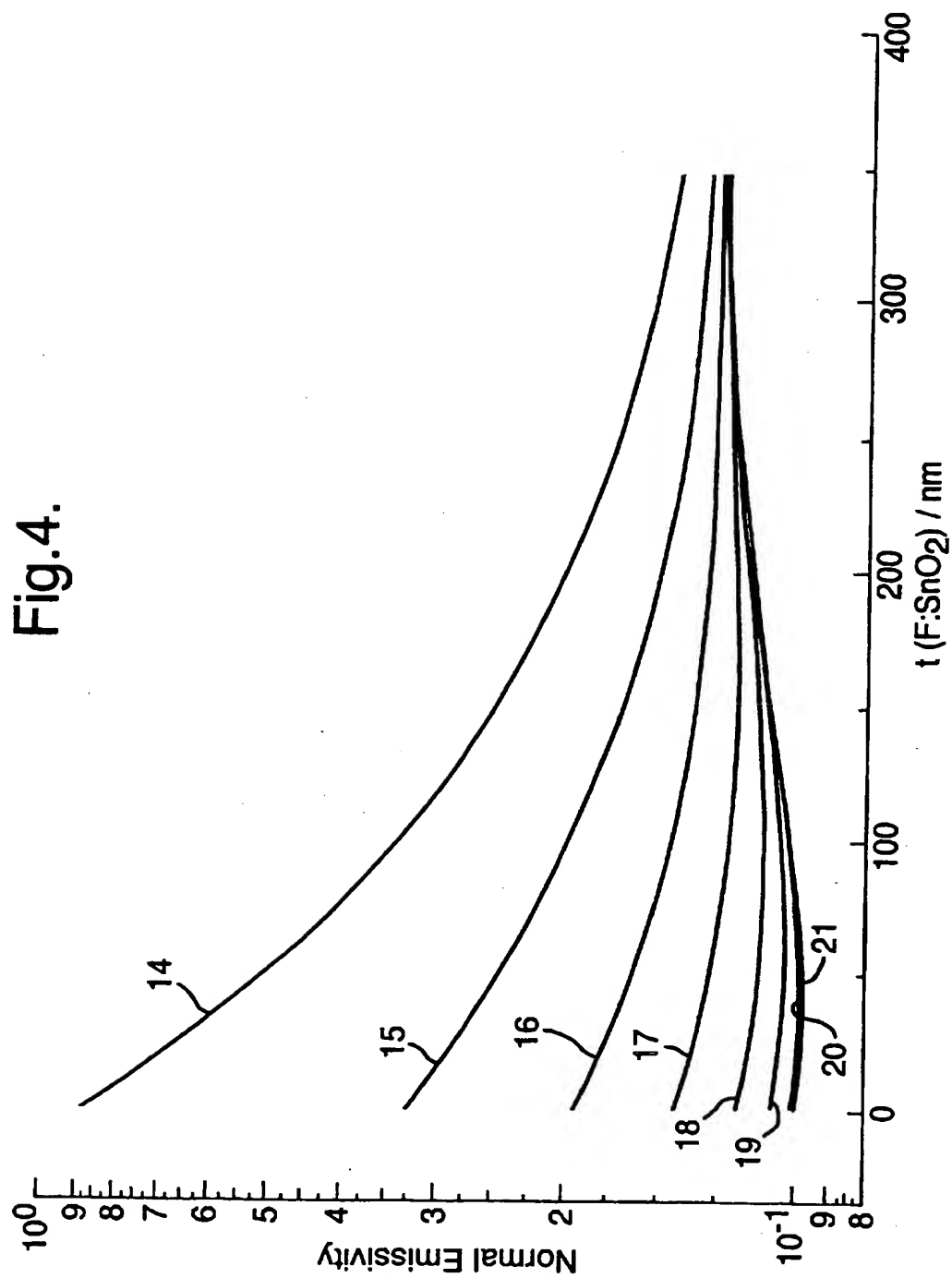
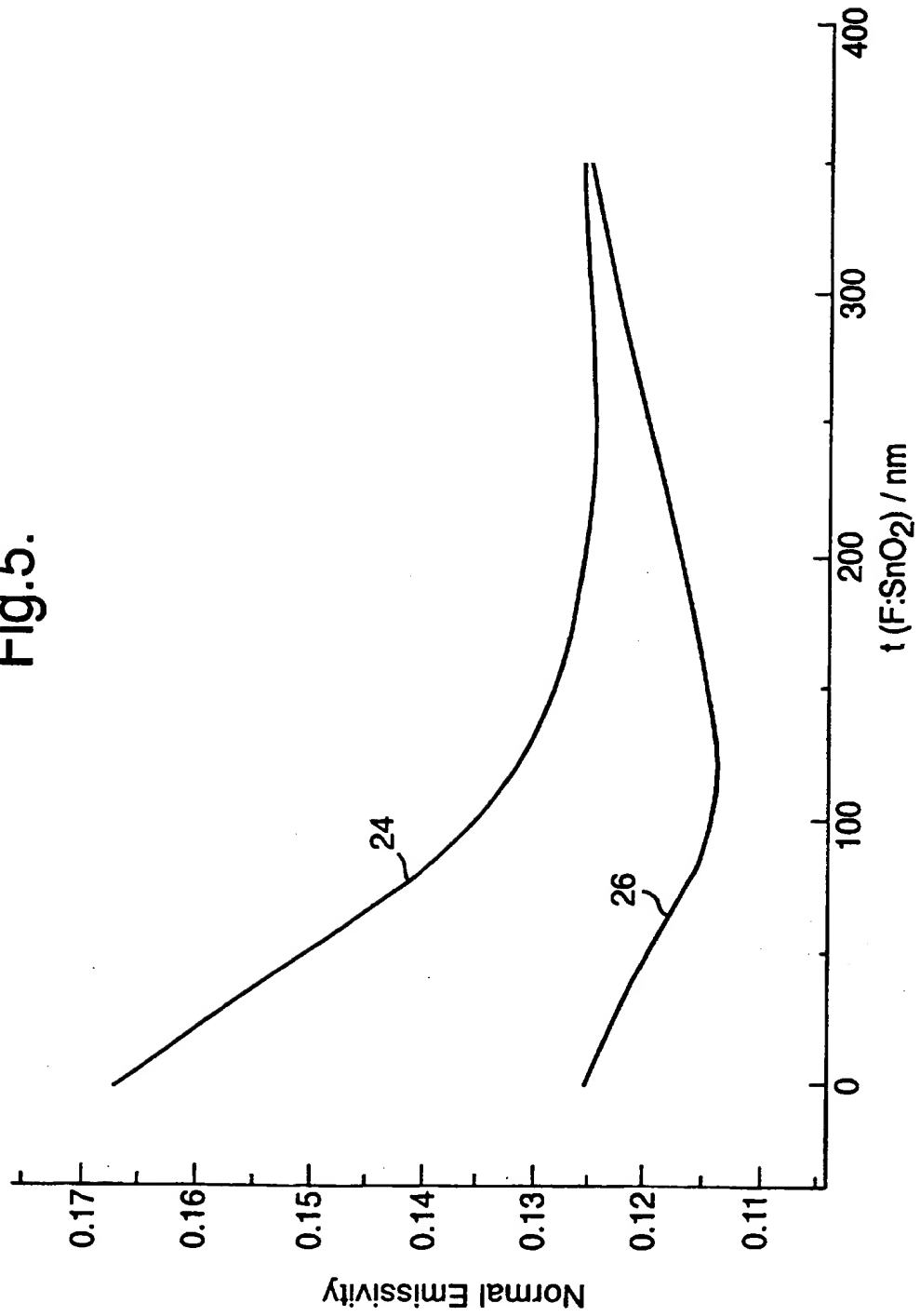


Fig.5.



Coated Glass

This invention relates to coated glass and in particular to low emissivity coated glass.

There has been interest in the production of low emissivity coatings on glass for use in buildings and in other applications. Low emissivity coatings on glass reflect infrared radiation, reducing heat loss through the glass and so improving the insulation of windows. Low emissivity coatings are useful not only because of cost benefits associated with better insulated windows but also because regulatory authorities are pressing for lower emissivity glazings.

One of the most successful low emissivity coatings on glass has been a coating with a functional layer of the transparent conductive metal oxide, doped tin oxide. For example, the applicant's product, Pilkington K Glass (Pilkington K Glass is a trade mark of the applicant) has a coating comprising a layer of fluorine doped tin oxide. Pilkington K Glass has an emissivity typically of 0.155 with high visible light transparency. Other coated glasses with coatings comprising transparent conductive metal oxides are disclosed in GB 2 031 756B. The invention of GB 2 031 756B is particularly directed to reducing iridescence in transparent conductive metal oxide coatings on glass by means of anti-iridescent layers in the coating. Although other transparent conductive metal oxides are disclosed in GB 2 031 756B (indium oxide and cadmium stannate), fluorine doped tin oxide is the preferred transparent conductive metal oxide.

Generally, fluorine doped tin oxide layers are preferred in the production of low emissivity coated glasses for reasons of performance and cost. To achieve lower emissivity, thicker coatings of transparent metal oxides are applied because the emissivity of such a coated glass tends to fall with increasing thickness of the coating until a limiting value of emissivity is reached, the intrinsic properties of the doped metal oxide progressively limiting the improvement in performance as the

thickness of the doped metal oxide (functional) layer is increased. Very thick coatings of fluorine doped tin oxide also tend to have high haze, especially when applied on-line by chemical vapour deposition during the float glass production process, which is aesthetically unsatisfactory and hence commercially undesirable. Taken together these two factors mean that the emissivity of generally commercially available fluorine doped tin oxide coated glasses is typically above 0.15. There is an objective to reduce emissivities to or below 0.12.

There are other known transparent conductive metal oxides suitable for forming low emissivity coatings on glass for example doped zinc oxides, other doped tin oxides and indium tin oxide, i.e. indium oxide doped with tin, also known as ITO. ITO is of particular interest because a single layer of ITO allows the production of coated glasses with emissivities below 0.12 and even below 0.1. Unfortunately indium is an expensive metal and the thicknesses of ITO layers on glass that are required in order to reduce emissivity to the required levels result in prohibitive cost for general architectural use.

Methods are known for the production of ITO coatings on glass, and for the production of ITO and other doped and also undoped indium oxide coatings on glass. For example, United States patent 4 286 009 relates to a composite coating for the absorber surface of a solar heat collector. Each of the two layers of the composite coating is of metal oxides selected from the group consisting essentially of oxides of tin, antimony, indium and iron. The invention is for solar energy absorbers and so is not related to the production of transparent low emissivity coated glass, but a method for the production of indium oxide coatings, doped with tin oxide, is described, the coatings being deposited by spraying a solution of indium (III) chloride and stannic chloride in ethyl acetate on to hot glass at 650°C.

There have been other attempts to produce ITO coatings on hot glass. For example European patent application EP 0 192 009A2 relates to the formation of indium oxide coatings on hot glass and is particularly concerned with the application of an indium oxide precursor to a hot glass ribbon on its exit from the float bath, for example at a glass temperature of 600°C. Indium oxide precursors disclosed are acetylacetonates of indium (in solution) and indium formate either in solution or in powder form.

Additionally, it is well known in the glass coating art that ITO and other metal oxides can be deposited as coatings by sputtering including reactive sputtering.

Unfortunately, single layers of transparent conductive metal oxides either are not able to produce coated glasses of sufficiently low emissivity or have other problems, for example high cost or high haze.

In addition to coatings of doped and undoped indium oxide, coatings comprising two or more layers of indium oxide, each layer doped with a different amount of tin, are known. United States patent US 4 507 547 discloses a heat wave shielding lamination (by which is meant a multi-layer coating) consisting of two types of infrared shield layers having different optical characteristics, both types of infrared shield layers being indium tin oxide but with different amounts of tin. The infrared shield layers have approximately the same refractive indices in the visible light range so as to obtain high visible light transmittance but have different refractive indices in the infrared spectrum against which shielding is required. The layers exhibit a shielding effect against infrared rays by interferential reflection. This is achieved by setting the optical thicknesses of each layer at $\lambda/4$ (the optical thickness of a layer is the product of the physical thickness and refractive index), λ being chosen as the wavelength at the centre of the infrared spectrum.

Unfortunately, although the multiple layer system described in US 4 507 547 based on indium tin oxide with layers differing in degree of tin doping is able to produce low emissivity coated glass it does not overcome the problem of the high cost of indium oxide coated glass products.

The applicants have now found that a desired low emissivity may be achieved with a coating suitable for application on line to hot glass without relying wholly on an expensive indium oxide for the desired low emissivity and without the need for an excessively thick coating likely to lead to serious problems of haze. The applicants have achieved this by using a coating comprising two layers of doped metal oxide each having a different host metal oxide.

According to an aspect of the present invention a low emissivity coated glass comprises a glass substrate with a coating comprising a first layer of a doped metal oxide in combination with a second layer of a doped metal oxide characterised in that the host metal oxides in each of the said layers are different.

The glass substrate for coating may be, for example, a soda lime silicate glass produced by the float glass process, or any other type of glass for which a low emissivity coating would be useful.

The host metal oxides of each layer are preferably based on different metals, although they may be based on the same metals of different oxidation state or metal oxides of distinctly different structures.

Ideally, the first layer of a doped metal oxide comprises a layer of doped tin oxide. This is advantageous because methods to produce such doped tin oxide layers are known, suitable precursor materials are readily available, and the resultant doped tin oxide layers have advantageous properties of emissivity and durability. One method to produce doped tin oxide

layers is chemical vapour deposition (CVD) wherein a vapour of a source of tin, for example tin tetrachloride, and a dopant source are contacted on a substrate as described in, for example, GB 2 026 454B. Other methods are sputtering from a tin or tin oxide target, and "wet" methods for example sol gel coating. These methods for applying layers of tin oxides and doped tin oxides are well known in the glass coating art.

The second layer of a doped metal oxide preferably comprises a layer of indium tin oxide. The advantage of this aspect is that low emissivities (significantly lower than readily achievable with doped tin oxide without unacceptable haze) may be achieved even though the thickness of the indium tin oxide layer is lower than would be required for a low emissivity coating comprising only an indium tin oxide layer. Methods for applying layers of indium tin oxide are well known in the glass coating art.

The properties of coatings of indium tin oxide depend on the method used to apply the coatings, and the nature of the substrate. Usually, indium tin oxide coatings prepared by chemical vapour deposition have higher electrical resistivities than those prepared by sputtering. Higher resistivities are associated with higher emissivities and a typical resistivity of a chemical vapour deposited indium tin oxide coating is 2.15×10^{-4} ohm cm compared to a typical resistivity of a sputtered indium tin oxide coating of 1.5×10^{-4} ohm cm.

Alternatively the second doped transparent conductive metal oxide may be doped zinc oxide. Although more difficult to achieve emissivities as low as with an indium tin oxide layer, the use of doped zinc oxide has the advantage of reducing costs still further.

Preferably, the second layer is overcoated by a metal oxide layer. Thus, in the preferred aspect where indium tin oxide is used as the second layer, the metal oxide layer overcoats the

indium tin oxide layer that is, the indium tin oxide layer would be disposed between the glass surface and a metal oxide layer. A particular advantage of this is that the second layer is protected from degradation during processing after it is applied including subsequent toughening or bending. Preferably the metal oxide layer is the first doped metal oxide layer.

In a preferred aspect of the invention the second layer has a thickness of between 50 nm and 300 nm, in a more preferred aspect a thickness of between 50 nm and 200 nm and in the most preferred aspect a thickness in the range 100 nm to 140 nm. This is advantageous because where, for example, the second layer comprises ITO and the first layer doped tin oxide there is a minimum in a graph of normal emissivity against thickness of the doped tin oxide layer (for a fixed thickness of ITO layer) which is particularly distinct for values of ITO layer thickness of 50-300 nm, especially 50-200 nm and doped tin oxide layer thickness of under 250 nm, especially under 200 nm.

The thickness of the first doped metal oxide layer may be between 20 nm and 200 nm preferably between 50 nm and 200 nm and especially between about 50 nm and 150 nm. This achieves the advantage that the haze that sometimes is produced in a coating, especially in CVD coating processes, is minimised because the haze tends to increase with the thickness of the layer. When the second layer is overcoated by a layer with inferior low emissivity properties some of the second layer's infrared performance may be lost through absorption in the overcoat. Therefore, the emissivity of the stack could progressively increase with increasing thickness of the first layer. However, we have found that provided the second layer is thin, adding the first layer will improve emissivity.

The coating would normally comprise an additional layer or additional layers whereby the migration of alkali ions may be blocked. Glass surfaces contain alkali ions that can migrate into coatings. The migration of alkali ions into doped transparent conductive metal oxides can result in the degradation of the conductivity and an increase in the emissivity of the coating. It is therefore advantageous to have layers disposed directly on the glass surface, or at least between the glass surface and the functional layers, that reduce or eliminate the migration of alkali ions. These layers may be composed of silicon oxide, silicon oxycarbide or silicon oxynitride or other metal oxides especially aluminium oxides, titanium oxide, indium oxide and multicomponent oxide coatings containing these oxides.

Usually the coating would comprise an additional layer or additional layers whereby the optical properties of the coating may be adjusted. Such layers include anti-iridescent layers as disclosed in, inter alia, GB 2 031 756B and also anti-reflection layers. This is advantageous because it allows coated glass to be produced that has neutral colour in reflection and/or transmission or reflection and transmission colours of particular values. Additionally this aspect allows the reflectivity of the coated glass to be reduced and the transmission increased.

With appropriate layer positioning and selection of layer material, the same layer or layers may both be anti-iridescent and a barrier to alkali metal ion migration from the glass, see for example European patent specification EP 0 275 662.

Preferably the emissivity of the coated glass is less than 0.15, more preferably the emissivity of the coated glass is less than 0.12.

A preferred dopant for the doped tin oxide layer is fluorine. This is advantageous because it is known that fluorine doped tin oxide can be commercially produced by coating the hot glass

surface during the float glass production process, and is cheap, effective and the fluorine dopant is readily incorporated in tin oxide.

Coated glass according to the invention may be incorporated in a multiple glazing unit with the pane of coated glass in spaced opposed relationship to a second glazing pane.

Another aspect of the invention is a method of producing coated glass. The layers of ITO, doped tin oxide or other metal oxide layers may be deposited by known techniques, for example by sputtering, including reactive sputtering or by pyrolysis, for example CVD. Indeed, it is an important advantage of the invention that the layers are susceptible to deposition by pyrolytic techniques providing for the possibility of applying the coating to the hot glass ribbon during the production process.

In order that the present invention may be more readily understood, reference will now be made to the accompanying drawings, in which:

Figure 1 illustrates schematically and in section a coated glass according to the invention, the coating comprising two layers,

Figure 2 illustrates schematically and in section a coated glass according to the invention, the coating comprising two layers and additionally plural anti-iridescent and alkali blocking layers.

Figure 3 illustrates schematically and in section a coated glass according to the invention, the coating comprising two layers, plural anti-iridescent and alkali blocking layers and additionally an anti-reflection layer.

Figure 4 illustrates in graphical form the logarithm of the simulated normal emissivity of coated glasses of the type illustrated in Figure 1, where the layer 4 is of ITO and the

layer 6 of fluorine doped tin oxide. Each curve represents the emissivity for a fixed thickness of ITO layer, 4, as a function of varying thickness of the fluorine doped tin oxide (F:SnO_2) layer, 6.

Figure 5 illustrates in graphical form the simulated normal emissivity of a coated glass as in Figure 4 except that the emissivity axis is linear and the two curves illustrated are not among those shown in Figure 4.

Referring to Figure 1, it is seen that a coated glass 1 comprises a glass substrate 2 and a coating, 3, comprising two layers, 4 and 6. The layers are an indium tin oxide layer, 4, of thickness 175 nm and a fluorine doped tin oxide layer, 6, of thickness 100 nm. The normalised emissivity of this coated glass would be 0.113 as produced by chemical vapour deposition.

Figure 2 illustrates a coated glass with layers of ITO, 4, and fluorine doped tin oxide, 6, but additional under-layers as follows: 10 is an undoped tin oxide layer and 8 is a layer of silicon oxide both as generally described in United Kingdom patent specification GB 2 115 315B. The underlayers act as anti-iridescent and alkali blocking layers.

Figure 3 illustrates a coated glass with the same layers as illustrated in Figure 2, with an additional anti-reflection layer, 12. The anti-reflection layer is a low refractive index material with an optical thickness of for example approximately one quarter of a wavelength in the centre of the visible spectrum, typically 550 nm. An effective anti-reflection coating material is silicon dioxide which has a refractive index of approximately 1.46. Other materials that may be suitable include magnesium fluoride. The principles that govern the optical properties of anti-reflection layers are well known, and other anti-reflection layers and stacks may also be used.

Figures 4 and 5 illustrate the effect on normal emissivity of variation in the thickness of the two layers according to the embodiment of the invention depicted in Figure 1. The normal emissivity of a coated surface is computed from its spectral reflectance curve measured at nearly normal incidence with an infrared spectrometer as described in International Standard ISO 10292 : 1994 (E), Annex A. Normal emissivity (ϵ_n) is defined in ISO 10292 : 1994(E), Annex A at 283 K as : $\epsilon_n = 1 - R_n$. The Drude model and matrix method were used to simulate the normal emissivity of coated glass by calculating the reflectance of a two layer stack in the wavelength region 5-55 μm . To obtain an overall value of normal emissivity the values at each wavelength were weighted by the intensity of black body radiation according to British Standard BS 6993. These values were integrated to give overall emissivity. The values of normal emissivity were calculated assuming properties of each layer consistent with their production using CVD. Each curve depicts normal emissivity at fixed thickness of the ITO layer, 4, as a function of the thickness of the doped tin oxide layer, 6. The thicknesses of the ITO layers for the curves in Figure 4 are as shown in Table 1.

Table 1

Curve	Thickness of ITO layer/nm
14	0
15	50
16	100
17	150
18	200
19	250
20	300
21	350

In Figure 4, for thin ITO layers of under 150 nm overcoated with layers of up to 300 nm of F:SnO₂ emissivity is reduced. However, for thicker ITO overcoating with up to 100 nm of F:SnO₂ results in a small reduction in emissivity, but emissivity is actually increased when the ITO is overcoated with more than 100 nm of F:SnO₂. The increase in emissivity on overcoating with F:SnO₂ becomes increasingly more apparent as the thickness of the ITO layer is increased and for ITO thicknesses of 300 nm or more there is little benefit from overcoating with even small thicknesses of F:SnO₂.

For the embodiments whose emissivity is illustrated in Figure 4 the best low-emissivity performance was obtained using a thick ITO layer overcoated with a thin F:SnO₂ layer. However, for cost effectiveness it is desirable that the ITO layer of a low emissivity coated glass product be as thin as possible because of the high cost of indium.

Figure 5 illustrates two further curves at thicknesses of the ITO layer of 117.5 nm, 24, and 175 nm, 26. The surprising nature of the applicant's invention is clearly shown by curves 24 and 26, especially curve 26, where a minimum in normal emissivity is apparent at a thickness of the doped tin oxide layer in the preferred range of 50 nm to 150 nm at a value of 115-125 nm. Even when the thinner ITO coating of curve 24 is used, little additional benefit is achieved by increasing the thickness of the doped tin oxide layer above 150 nm. These thicknesses are likely to be in the region of cost effective embodiments of the present invention.

The invention is illustrated but not limited by the following Examples.

In the Examples the Drude method was used to calculate normal emissivity in a similar way to the method described for the generation of the data for Figures 4 and 5.

The reflectance is governed by the carrier concentration, carrier mobility and relative thickness of each layer. The carrier concentration (n) and carrier mobility (μ) depend on the chemistry and method of production of the coating layer. Empirical values of n and μ for sputtered and CVD produced ITO and CVD produced fluorine doped tin oxide layers are reproduced in Table 2. The tabulated values of n and μ were determined from laboratory samples of CVD and sputtered ITO and commercially produced layers of CVD fluorine doped tin oxide by the usual methods. In the simulation of normal emissivity it was assumed that there would be no deterioration in performance due to alkali ion migration. In practice, alkali ion migration from the glass substrate would be prevented or reduced by additional alkali blocking layers in the coating.

Table 2

	Sputtered ITO	CVD ITO	CVD Fluorine doped tin oxide
Carrier Concentration n / cm^{-3}	8.45×10^{20}	8.57×10^{20}	3.89×10^{20}
Carrier Mobility $\mu / \text{cm}^2 \text{V}^{-1} \text{s}$	50.0	33.85	37.07
Effective Electron Mass	-	0.25	0.30

Examples

The normal emissivity together with the thickness of the coating layers in Examples 1-5 are shown in Table 3. In the Examples the normal emissivity of a coating stack of the type illustrated in Figure 1 comprising a layer of ITO, 4, overcoated with a layer of fluorine doped tin oxide, 6, was calculated. In Example 1-4 the properties of the layers were those typically seen in CVD ITO and F:SnO_2 with values of n and μ as shown in Table 2.

Example 5 is a stack with layers of the same thickness as Example 1, but the ITO layer has values of n and μ commonly seen in sputtered ITO coating layers with consequent improvement in the normal emissivity of the coated glass over Example 1.

Table 3

Example	Thickness of doped tin oxide layer / nm	Thickness of indium tin oxide layer / nm	Normal Emissivity
1	100	175	0.113
2	157.5	117.5	0.126
3	50	300	0.098
4	100	250	0.104
5	100	175	0.097

Claims

1. A low emissivity coated glass that comprises a glass substrate with a coating comprising a first layer of a doped metal oxide in combination with a second layer of a doped metal oxide characterised in that the host metal oxides in each of the said layers are different.
2. A coated glass according to claim 1 wherein the first layer of a doped metal oxide comprises a layer of doped tin oxide.
3. A coated glass according to claim 1 or 2 where the second layer of a doped metal oxide comprises a layer of indium tin oxide.
4. A coated glass according to claim 1 or claim 2 wherein the second layer of a doped metal oxide comprises doped zinc oxide.
5. A coated glass according to any preceding claim wherein the second layer is overcoated by a metal oxide layer.
6. A coated glass according to any preceding claim wherein the second layer has a thickness in the range 50 nm to 200 nm.

7. A coated glass according to any preceding claim wherein the second layer has a thickness in the range 100 nm to 140 nm.
8. A coated glass according to any preceding claim wherein the first layer has a thickness in the range 20 nm to 200 nm.
9. A coated glass according to any preceding claim wherein the first layer has a thickness in the range 50 nm to 200 nm.
10. A coated glass according to any preceding claim wherein the first layer has a thickness in the range 50 to 150 nm.
11. A coated glass according to any preceding claim wherein the coating comprises an additional layer or additional layers whereby the migration of alkali ions may be blocked.
12. A coated glass according to any preceding claim wherein the coating comprises an additional layer or additional layers whereby the optical properties of the coating may be adjusted.
13. A coated glass according to any preceding claim wherein the emissivity of the coated glass is less than 0.15.

14. A coated glass according to any preceding claim wherein the emissivity of the coated glass is less than 0.12.
15. A coated glass according to any preceding claim wherein the first layer is of tin oxide doped with fluorine.
16. A multiple glazing unit comprising a pane of coated glass as claimed in any of the preceding claims in spaced opposed relationship with a second glazing pane.
17. A method for producing a low emissivity coated glass comprising applying a coating on to a glass substrate the coating comprising a first layer of a doped metal oxide and a second layer of a doped metal oxide, the host metal oxides of each of the said layers of the coating being different.
18. A coated glass substantially as herein described with particular reference to any of the Examples 1-5.
19. A coated glass substantially as herein described with reference to and as illustrated in any of Figures 1 to 5 of the drawings.



Application No: GB 9804907.5
Claims searched:

Examiner: Pete Beddoe
Date of search: 27 May 1999

Patents Act 1977
Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.Q): C7F (FBAL, FBAX, FBBL, FBBX, FBXL, FBGXX, FHB, FHD, FHE, FHX, FPCL, FPCX, FPD, FPD, FPD)

Int Cl (Ed.6): C03C 17/34; C23C (14/08, 16/40)

Other: Online: WPI, EPODOC, JAPIO

Documents considered to be relevant:

Category	Identity of document and relevant passage	Relevant to claims
X	EP 0811583 A1 (NIPPON) see esp Table 14	1,17 at least
X,E	WO 98/11031 A1 (PILKINGTON) 19 March 1998 see esp exs 1,2	1,3,17 at least
X	US 5776603 (SAINT-GOBAIN) see esp col5 lines 1-18 & lines 23-26; col6 lines 55-58 & reference example 3	1,2,15,17 at least
X	US 5756192 (FORD) see esp claim 1 & examples	1,2,15,17 at least
X	US 5348805 (SAINT-GOBAIN) see esp exs 4 & 11	1,2,15,17 at least
X	US 5342676 (SAINT-GOBAIN) see esp col6 lines 14-19 in conjunction with exs 2-4	1,2,15,17 at least
X	US 5244692 (SAINT-GOBAIN) see esp ex 9	1,2,15,17 at least
X	US 4507547 (KABUSHIKI) see esp ex 4 & fig 7	1,17 at least
X	US 4419386 (GORDON) see esp example 1	1,2,15,17 at least

X Document indicating lack of novelty or inventive step
Y Document indicating lack of inventive step if combined with one or more other documents of same category.

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A Document indicating technological background and/or state of the art.
P Document published on or after the declared priority date but before the filing date of this invention.
E Patent document published on or after, but with priority date earlier than, the filing date of this application.



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18.



INVESTOR IN PEOPLE

Application No: GB 9804907.5
Claims searched:

Examiner: Pete Beddoe
Date of search: 27 May 1999

Category	Identity of document and relevant passage	Relevant to claims
X	US 4206252 (GORDON) see esp example 1	1,2,15,17 at least

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.